



**adolphe merkle institute**  
excellence in pure and applied nanoscience

UNIVERSITY  
OF FRIBOURG  
SWITZERLAND

## Focused ion beam

### Introduction

Version 1 – September 2020

*IN SITU* LAMELLAE / AUTO TEM





## ***Universal rules***

**Rule 1: don't touch a control if you are not sure of the outcome of that action**

**Rule 2: never, ever force anything beyond finger strength**

**Rule 3: wear gloves when touching anything that goes into the chamber**

**Rule 4: if in doubt, ask for help**

Demonstration: Loading the halfmoon grids.....	6
Demonstration: mounting the row bar .....	8
Demonstration: UI setup .....	9
Demonstration: Sharpen the easyLift tip.....	10
Demonstration: Start the AutoTEM software .....	11
Demonstration: Creating a new project .....	12
Demonstration: Chunk mill.....	14
Demonstration: Lift out .....	18
Demonstration: Thinning.....	20

## Demonstration: Loading the halfmoon grids

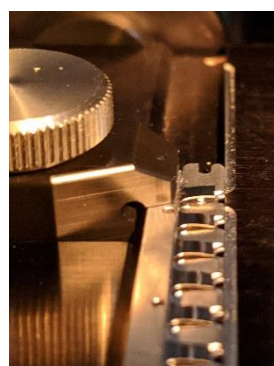
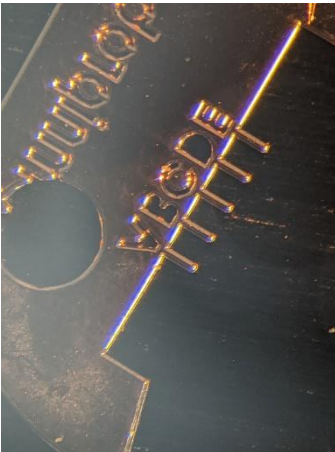
### Prerequisites:

- Running xT server
- Running UI
- Stage vented

### Action:

- Loading the grids on a row bar

Halfmoon grids are the adapter between SEM and TEM



The halfmoon grids have two sides:

- Shiny side (=should be down)
- Matt side (=should be up)

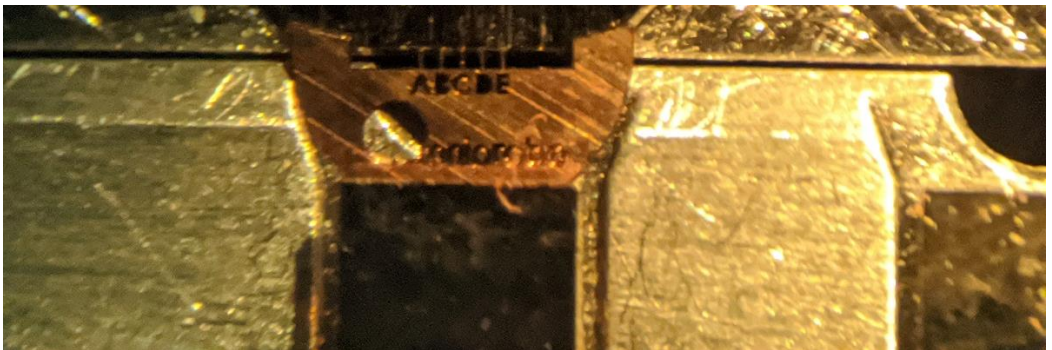
The row bars also have two sides:

- Bottom side: with holes in the center
- Upper side: with flat clamps



### Experiment: load a halfmoon grid in the row bar

- Place 2-3 halfmoon grids on the stage, matt side up
- Place the row bar with one hole over the stage pin. The upper side should align with the edge of the front panel of the stage
- Place the stage screw over the row bar and tighten it. This will lift the clamp of the row bar.
- gently guide the halfmoon grid towards the position with the opened clamp. Make sure the shiny side is down. Steer the round side of the grid under the clamp.
- Release the screw. The clamp releases and holds the grid.
- Move to the row bar to the next clamp if needed



**Demonstration: mounting the row bar****Prerequisites:**

- Running xT server
- Running UI
- Stage vented
- Row bar with half moon grids

**Action:**

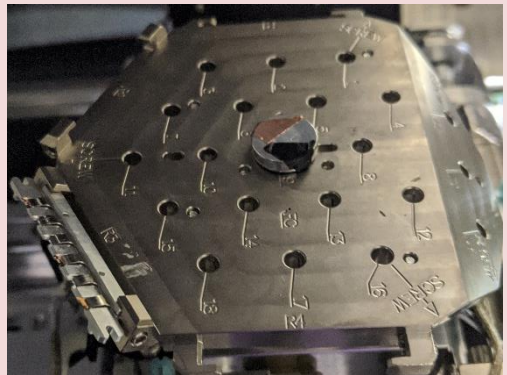
Mounting the row bar on the FIB SEM stage

The correct mounting of the row bar is critical

Put on gloves when handling the row bar.

**Experiment**

- Rotate the stage (using UI) 90°
- Open the stage and open the side screw of R3. This is the pre-tilted slot (pre-tilted to 38°). Place the row bar in R3 with the clamps facing up. The gently tighten the screw.
- Load your sample. Ideally, place only one stub in the middle of the stage
- Pump the stage and reset the stage rotation to 0°



The pre-tilted row bar will assure that the FIB-SEM beam is parallel to the sides of the chunk.



**Demonstration: UI setup****Prerequisites:**

- Running xT server
- Running UI
- Stage vented
- Row bar with half moon grids

**Action:**

Setup the software for autoTEM

**Do not skip these steps!**

**To run a successful AutoTEM project it is important to:**

**Experiment: setup the system**

- During pumping: home the stage
- When the vacuum allows, move the stage up, focus and link. Repeat until the sample (not the half moon grids) is at 7 mm eucentric height. Set eucentric height (CTRL+f) and repeat the 7mm setting.
- Reset all the beam shifts
- Take a nav-cam picture.
- Save the position of your region of interest
- Start the ion beam. Tilt to 52° and setup coincidence point. Link the ion beam image to the SEM image.
- Goto the half moon grids (R3, pre tilted) and pick a grid and a finger. Save the position

**Demonstration: Sharpen the easyLift tip****Prerequisites:**

- Stage pumped
- Samples loaded and coincidence point set
- Stage at 0° tilt
- e- beam and Ga ion beam on

**Action:**

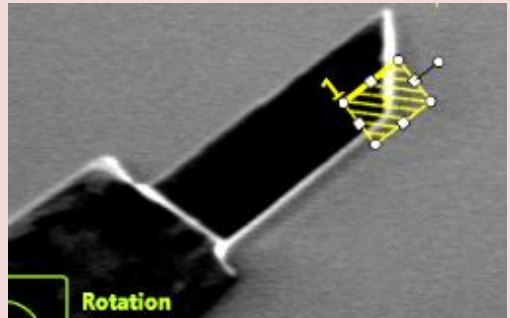
- Create a proper easyLift tip

Part of the tip will not survive

**Assure the stage is linked and LOWERED (15-20 mm)**

**Experiment**

- Insert the easy lift needle. Focus on the needle (around 7 mm) using the e-beam
- Image the needle with the Ga-ion beam and zoom in on the tip
- if needed cut away material at the lower base of the needle
- Rotate the needle 90° and repeat the Cutting
- This should yield a tip of about **2 μm**



**width and 1 μm height** (NOT LESS!). The tip should be oblique (as in the picture), with the longer part the upper part (in the ion beam image).

- Retract the needle.

## Demonstration: Start the AutoTEM software

### Prerequisites:

Running xT server  
Running UI  
Sample and half moon grid loaded  
Stage linked and focused

### Action:

Starting the AutoTEM

AutoTEM is the software that automates the lamellae making

Using the FEI menu, select AutoTEM 4.1

AutoTEM 4 has 3 tabs at the startup:

**Projects**      Projects from the past are saved here

**Templates**      Allows to save templates and change settings

**Alignment**      For technicians only

Note: the software can take some load of the procedure, but is not very robust. However, it helps to structure your steps.

**Demonstration: Creating a new project****Prerequisites:**

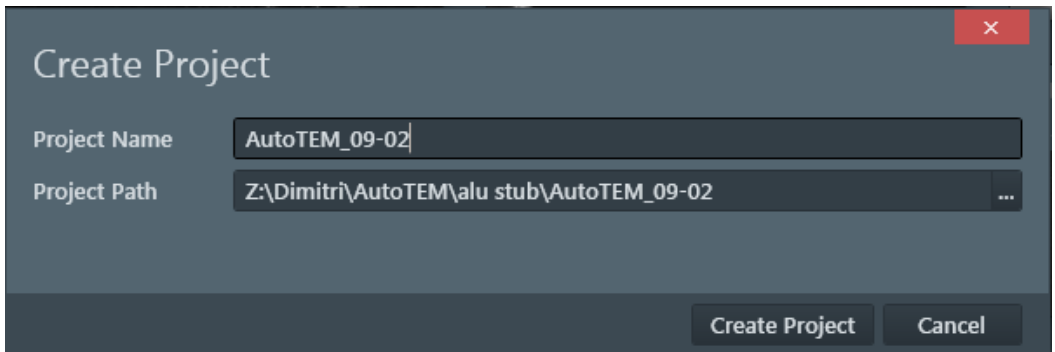
AutoTEM 4 running

**Action:**

Creating a AutoTEM project

Make sure you save on SharedData

Click the big + in the tab projects.



**Create Project**

Project Name

Project Path

**Create Project** **Cancel**

In the project path: make sure you save your data on Z (the shared data folder).

**Experiment: setup the project****Site list**

You can batch create lamellae sites. Add more if needed. The sites will be created in series.

### **In-situ lift-out**

This means the creating of lamellae. Here, you can select the relevant templates

(ex-situ lift-out and cross section are irrelevant)

### **Chunk position**

Here, you can set the position of the area of interest where the chunk (=rough lamellae) will be created.

- Imaging is done with the **Ga ion beam** / ETD dectector
- Assure you are at eucentric height, 52 degrees tilting and that the E beam and ion beam are linked
- Magnification is calculated (and applied) based on the chunk size you want to use!!

### **TEM grid position**

- **This part is critical**
- Rotate the stage -150 degrees (stage is on the right of the electron lens)
- tilt 52 degrees
- Goto the position of the finger (select from your saved position list) of the half moon grid.
- In the SEM image, you see the finger front on (52 degrees + 38 pretilt = 90 degrees)
- Focus, Link and set 7mm for the tip of a finger.
- In the TEM grid position, the imaging is done with the **Ga ion beam** / ETD detector

**Demonstration: Chunk mill****Prerequisites:**

AutoTEM 4 running  
 Project created and templates assigned

**Action:**

Automatically digging a chunk

**Try the multi site option!**

Click “chunk mill” at the bottom. This algorithm will automatically:

1. Create a fiducial marker at the set position
2. Create a protective Pt layer over your region of interest/chunk (future lamellae)
3. Ground (optional): deposits a thin layer of Pt across the site to reduce charging
4. Do the rough milling: create two trenches on both sides of the chunk
5. Create needle trench: on the left side of the chunk, material is removed to facilitate the needle attachment
6. Medium milling: this is the thinning of the trench at lower currents.
7. Cutout: creating a side and undercut. After this step, the chunk is only attached on the right side with a short bridge
8. Cleanup: overtilting the chunk to remove debris at the lower end of the chunk
9. Create a thinning fiducial (aka Baby fiducial): create a small fiducial on the right side of the chunk, for alignments at the tips.

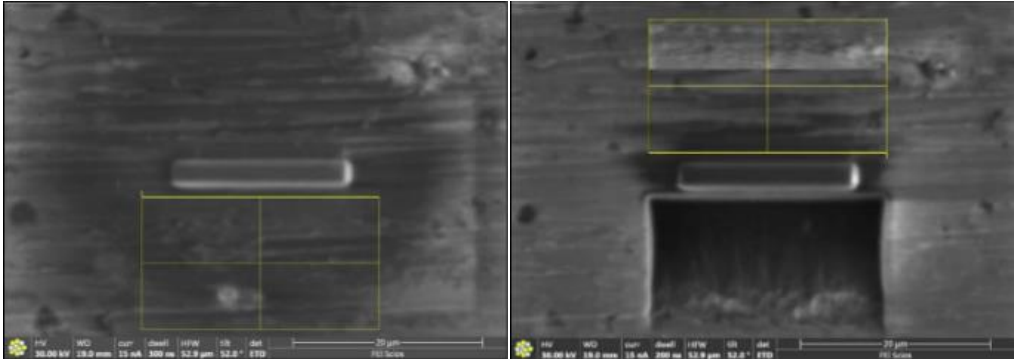
This entire procedure should run automatically and takes about 30 minutes of time per site.



**Experiment: Manual procedure**

1. Find area of interest
2. Set coincidence point using e-beam + Z-adjustment (start with: zero beam shift and uncheck Z-Y link)
3. Optional: e-beam deposition
4. Tilt to 52° and cover the area of interest with Pt by Ga ion.
5. Rough cut/bulk milling using Regular Cross Section + Si multipass:
  - Regular Cross Section. Size of pattern: X slightly wider as Pt layer width
  - Set Z to required depth , Y=2 times Z.
  - Choose an ion beam current according to size and material (usually 5-50nA).
  - Put the pattern not too close to the Pt layer, not overlapping with the Pt layer
  - Start to mill front side first.
6. Repeat from the other (back) side
  - Rotate pattern 180°
  - NOTE: end point (where patterning stops = the thick yellow line) is always the Pt layer
7. Finally: apply an extra tilt (in + and -) using the cleaning cross section.
  - Z = ½ of the bulk milled depth
  - Apply to the front and backside

The final lamella thickness: 1-1.5 µm



Next step: make the needle trench. The needle trench is a small gap on the left side of the chunk to allow space for the EasyLift needle. Usually, the automated routines take care of this.

Next step: The overcut or J cut. This will disconnect almost the entire chunk, leaving it connected to the bulk with only a small bridge. Depending on drift or the precision of the beam coincidence setting, the system may have troubles finding back the region of interest.

### Experiment: create the cut-out

Cut-out: Tilt to 7 degrees (make sure your beam coincidence is set perfectly).

#### First rectangle: at the bottom (3/4 from top).

- Width of bottom cut at about  $1\ \mu\text{m}$  width.
- Do not put this rectangle too deep (at about  $\frac{3}{4}$  from top). It defines the width of your final lamellae

#### Second rectangle: at the EasyLift side (usually left side)

- This side is to be cut out completely

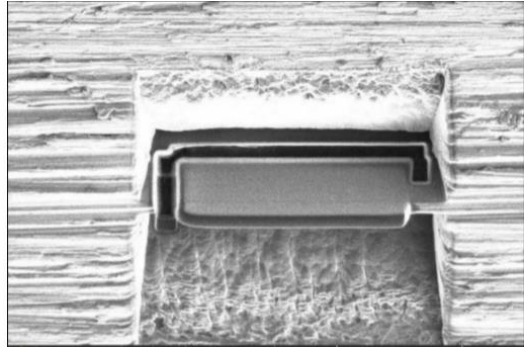
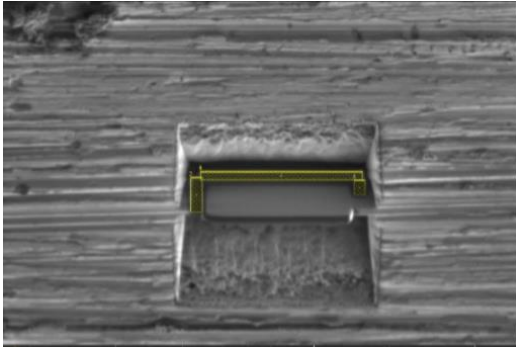
#### Third rectangle: at right side

- Leave a part of the lamella attached to the bulk.



**Milling settings:**

- Choose parallel milling (right top patterning page) this avoids redeposition
- $z=2\mu\text{m}$  (about the lamellae thickness)



**Make absolutely sure the J cut has passed through the entire lamella and is only attached with the bridge**

**Demonstration: Lift out****Prerequisites:**

Chunk created

Half moon grid in row bar R3, matt side up

EasyLift needle sharpened

**Action:**

Take the chunk out and move connect it to the half moon grid

**The tensesst steps of the entire procedure**

These steps are semi-automated: at the bottom right, a visual will appear to ask you to check if the desired situation is matched.

Check your half moon grid finger: in ion beam imaging, it should appear from top, you should see the bar on the upper side and at 0° stage tilting, you should look at the finger face on. Lower the stage to 10mm or more to allow space for the needle. The needle is visible in SEM and ion image at lower magnifications.

**Experiment: connect EasyLift needle**

Step 1: a series of image will be shown where you have to put an overlay on the image. This positions the tip of the needle in the 3D space inside the chamber.

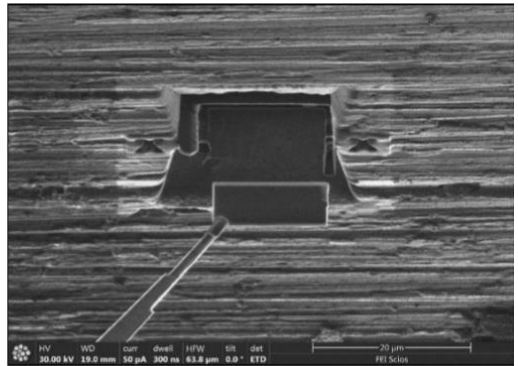
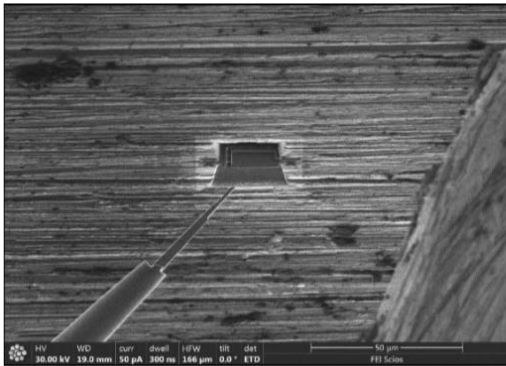
Step 2: The tip is inserted and is already very close to the lamellae. Align the tip on the left side of the lamella. Do not use “jog” until you are sure what you are doing!

Step 3: carefully align the tip as shown in the steps. You want to align it to the upper part of the lamella and get as close as possible.

Step 4: the needle is attached to the Pt layer of the lamella using Pt deposition.

Step 5: Wait for the vacuum to recover and cut the bridge that attaches the lamella to the bulk (on the right side). Now the lamella is only attached to the needle. The chunk can be extracted.

Note: you can now retract the EasyLift needle. The lamella will not get damaged.



### Experiment: Drive to grid and attach chunk to half moon grid

Note that the correct setup of the grid is paramount!

Step 1: you will again be provided with images and you need to move the overlay.

Step 2: position the lamellae parallel with the top of the grid

Step 3: GIS free welding (use 4 strips of  $4 \times 1 \mu\text{m}$  at about 1 nA). This step can be run only once: if it fails, do not repeat it. The idea is the mill part of the Cu grid and use the redeposition of the Cu to attach the lamella. Usually, this works very well.

Step 4: (optional) additionally weld the lamella with Pt deposition

Step 5: cut off the EasyLift needle: this will again yield a tip that is longer on the upper part than the lower part. Retract the needle.

**Demonstration: Thinning****Prerequisites:**

Lamella welded to the half moon grid  
 Ion beam looking perpendicular onto the lamella

**Action:**

Reduce the thickness from 1-1.5  $\mu\text{m}$  to 50-100 nm

**Know your material!**

In all cases: use cleaning cross sections, Tilt angle: 0 degrees (=38 degrees due to pretilt). Electron beam: 30 kV, ion beam 30 kV. The goal is to reach transparency using an electron beam at 2-5 kV

**Experiment: thinning****Imaging**

Quadrant top left: secondary (ETD)

Quadrant bottom left: STEM detector using bright field

Quadrant top right: ion beam (ETD)

→ Use 30 KV SEM to start with. Adjust to lower KV as the lamella becomes transparent in the STEM image. Transparency at 5-10 kV will make a thin enough lamella (tested on Al)

**Medium Thinning**

Step 1. (CCS, 1nA, 30 kV) Repeat the cleaning cross sections at  $+2^\circ$  and  $-2^\circ$  tilt angles

Step 2. (CCS, 300pA, 30 kV) Repeat at  $\pm 1.5^\circ$  tilt. You should yield a thickness of about 300nm

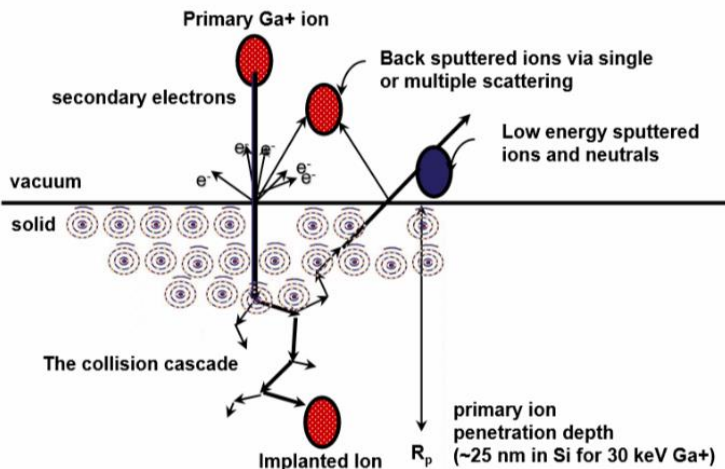
Step 3 (CCS, 100pA, 30 kV) Repeat at +/- 1.2° tilt

### Final thinning

For e-beam end-pointing use < 5kV. Low kV clean is always needed for crystalline material<sup>1</sup>

Step 4. (CCS):5 kV, 48 pA, +/- 5°

Step 5. (CCS):2 kV, 27 pA, +/- 7°



**For good quality TEM lamellae the amorphous layers need to be minimized**

<sup>1</sup> Milling @ 30kV: amorphous layer (in Si) = 23nm; @ 5kV: 6 nm; @ 2kV: 2 nm

- 
- Lamella from beam sensitive / soft samples
- 
- Lower the beam current (500pA and below) -> less melting -> more efficient milling
  - Lower ion dose
  - Use negative OL -150% and faster DT
  - Don't use e-beam while patterning
  - Sputter coat the sample
  - Total process will take longer; use autoTEM4 to prepare a chunk

NOTE: Temperature increase during milling; conductive material a few degrees (<10K), non-conductive material (low thermal conductivity) high temperature increases were measured ( $\text{SiO}_2 \pm 230\text{K}$ )

## Notes



**adolphe merkle institute**  
excellence in pure and applied nanoscience

UNIVERSITY  
OF FRIBOURG  
SWITZERLAND